

Section XVIII: Identification of Ketamine

I. Introduction:

Ketamine comes in a clear liquid and a white or off-white powder form. It is also found with other substances such as heroin and MDMA. Samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. Ketamine samples alone requires, by law, a test for the presence of Chloride Ion. This is done by a manual test for chloride ion or by IR.

II. Reagents:

- A.) 9:1 Methylene Chloride/Isopropanol or Ethanol
- B.) 2M Sulfuric Acid, H_2SO_4
- C.) Potassium Persulfate, $\text{K}_2\text{S}_2\text{O}_8$.
- D.) .2M Silver Nitrate, AgNO_3 .

III. Equipment:

- A.) Analytical balance
- B.) Weigh paper
- C.) Pipettes
- D.) Glass test tubes
- E.) 2 mL autosampler vials with Teflon caps
- F.) GC/FID: HP 6890 or 7890A
- G.) GC/MS: HP 7890A/5975C or HP 6890/5973 series.
- H.) Perkin Elmer Spectrum 100 FT-IR Spectrometer.

IV. Procedure:

A.) Chromatography by GC/FID and GC/MS.

1. Obtain gross weight of sample evidence bag.
2. Remove one sample packet from the evidence bag and weigh.
3. Record individual packet gross weight in logbook.
4. Remove powder from packet onto a weigh paper.
5. Reweigh the empty packet and record the weight.
6. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
7. Add powder back to packet and reseal.
8. Return packet(s) to original evidence back, reseal, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the powders net weight.

10. For up to 100 packets, analyze 10% of the total. Report the average of the individual the net weights.
11. For more than 100 packets, analyze the square root of the total. Report the average of all the individual net weights.
12. Add 1-2 mL of Ethanol or 9:1 Methylene Chloride/Isopropanol to the autosampler vial(s) containing the sample and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
14. GC/FID conditions are as follows:
Method: EXP.M
Oven:
 Initial Temp: 245°C
 Initial Time: 0.00 min.
 Rate: 10°/min.
 Final Temp: 290°C
 Run Time: 10 min.
 Max. Temp: 325°C
 Equilibration Time: 0.5 min.
Inlet:
 Mode: split (35:1)
 Initial Temp: 250°C
 Pressure: 24.99 psi
 Gas Type: Helium
Column:
 Capillary: HP-1 30m x 320um
 Initial Flow: 3.3 mL/min.
Detector:
 Temp: 300°C
 Hydrogen Flow: 30.0 mL/min.
 Air Flow: 400 mL/min.
 Makeup Gas: Helium
15. Obtain chromatographs. If ketamine is present, the instrument will detect a peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
16. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.
17. Place same sequence on the GC/MS autosampler and run.
18. GC/MS conditions are as follows:
Method: EXP.M

Oven:

Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.

Inlet:

Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

19. If ketamine is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance.

B.) Determining the presence of the Chloride Ion.

1. Analyst may either use the manual Test for Chloride Ion (see below) or the FT-IR Spectrometer.
2. Test for Chloride Ion:
 - a. Dissolve 3-5 mg of sample in 1 ml of 2M H₂SO₄. This will eliminate interference from non halides
i.e. CO₃⁻², CN⁻ and S⁻² etc.
 - b. Add 10-15 mg of K₂S₂O₈. This oxidizes Br⁻ and I⁻ but not Cl⁻ to Br₂ or I₂. A brown coloration indicates the presence of I₂ or Br₂. This will eliminate interference from I⁻ and Br⁻.
 - c. Heat to 100° C. for 5-10 minutes. This will drive off halides as gases Br₂ and I₂.
 - d. Cool then add 2 Drops of .2M AgNO₃. A white precipitate indicates the presence of Chloride (Cl⁻). AgF is soluble in water.

V. Results:

- A.) Record results of the GC/MS and Chloride Ion Test in logbook. Then transfer the results to appropriate evidence sheets that

came with the actual samples. Be sure to include date of analysis, net weight if applicable, number analyzed, results, and signature.

- B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.